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# (54) Delay charge and element, and detonator containing such a charge

Verzögerungsladung und Element, und Detonator mit solch einer Ladung

Charge à retard et élément, et détonateur contenant une telle charge

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## Description

### Technical Field

The present invention relates to a pyrotechnic delay charge for providing delays in the millisecond and second ranges.

### Background

Pyrotechnic delay charges are used in many fields, both military and civil, to provide a time delay between an inititating signal, for instance from an electrically activated fuse head or from a fuse, and triggering of a main reaction, such as ignition of a propulsive charge or triggering of a blasting charge. The charges will be described below in greater detail in relation to detonators for civil rock fragmentation.

A leading requirement for pyrotechnic delay charges is that they should burn with a well defined and stable burn rate having an insignificant time scatter. The burn rate should not be significantly influenced by the surrounding conditions or aging. Because of this, a burn having insignificant gas evolution and initial materials, intermediates and end-products with favourable and stable properties is required. The charges should be easily ignitable and provide good ignition transfer to other materials but without being excessively sensitive to jolts, vibration, friction or static electricity. The nominal rate should be adjustable with minor modifications of the charges, The charge compositions should be easy to prepare, dose and compress in safety. The charges should have a high energy content per unit weight and the incorporated components should not be too expensive.

Although conventional pyrotechnic elements can be said, in principle, to consist of a fuel and an oxidant, and therefore many substances should be usable, the above described requirements together significantly limit the choice of suitable initial materials. The component choice has come to be concentrated around a few established components within each field of application. For example, lead compounds are common ingredients in civil detonators.

Even though the amounts of pyrotechnic charge in the majority of initiator types are relatively small, there is a growing requirement that the charges should not contain toxic substances. This is in order to avoid problems during manufacture, to reduce emissions and also to lessen the problem of exposure at the point of end-use. It is also desirable that the preparation of the charges can be done without using solvents. Several components previously used in pyrotechnic elements are now no longer usable, for instance heavy metals.

A number of charges have been proposed with the goal of uniting good pyrotechnic properties with insignificant health consequences. For example, Swedish patent nos. 446 180 and 457 380 describe charges based on, inter alia, tin oxide as a principal non-toxic oxidant. However, these charges have less satisfactory properties as regards time adjustment and manufacture.

### The invention in general

A principal object of the present invention is to provide a delay charge which well fulfills the above general requirements for such charges. A particular object is to provide charges which have stable and reproducible burn times and suitable initial, intermediate and end-product properties. A further object is to provide a charge which does not contain toxic components. An additional object is to provide a charge which is water-insoluble, non-hygroscopic, which may be mixed or prepared in aqueous media, and which is also in other respects easy to handle and safe. Yet another object is to provide a charge which is energy dense and relatively cheap.

These objects are attained with the distinguishing features apparent in the patent claims.

In accordance with the invention, there is provided a delay charge comprising elemental silicon and bismuth oxide. These components are chemically stable, burn without substantial gas evolution and form stable residue products. The resulting delay periods are reproducible, linear and have insignificant scatter. The charges are easy to initiate, even without start charges. The components are entirely non-poisonous. The components are non water-soluble, non-hygroscopic and can be prepared in water. The components are easily handled and have a low price. Also, in other respects, the components exhibit suitable properties in the above-mentioned considerations.

Additional objects of the invention and the benefits attained will be apparent from the detailed description below.

### Detailed Description

The charge of the invention can be used for various pyrotechnic objectives, for instance as a start charge, firing charge or transfer charge but the main use is as a delay charge. A suitable burn rate for the charge of the invention is in the range of 10 to 200 mm/s, preferably between 15 and 150 mm/s and especially between 20 and 120 mm/s. For civil detonator applications, the charge is convenient for providing delays of the order of 10 to 3000 ms and especially between 20 and 2000 ms. These charges are hereafter referred to as "fast charges". The invention, however, is also suited to slower charges having burn rates in the range of 1 to 20 mm/s, and especially between 3 and 15 mm/s which are convenient for delays in the range of 0.5 to 10 seconds, especially 1 to 8 seconds. These charges are hereafter referred to as "slow charges". Primers and ignition charges may have burn rates above 150, especially above 200 mm/s.

Without limiting the invention to any theory of function or reaction, and especially not when more than the obligatory components are incorporated, the silicon component will be described below as a fuel component and the bismuth oxide component as an oxidant.

The silicon may be in the amorphous or preferably the crystalline form of the usual grade in the pyrotechnics context. The bismuth oxide is dibismuth trioxide ($Bi_2O_3$).

The relative amounts of silicon and bismuth oxide can be varied within wide limits. Mixtures which are stoichiometrically deficient in fuel may be used, especially for slow charges. A surplus of the fuel component relative to the oxidant is usually preferred. Under the premise that the silicon reacts to form silicon dioxide and the dibismuth trioxide is reduced to elemental form, a surplus of the silicon in relation to the stoichiometrically necessary amount (3:2) is preferred, preferably a mole ratio in excess of 2:1 or more preferably 3:1. The mole ratio should not exceed 6:1 and it is best not to exceed 5:1.

In absolute terms, it is preferred that the charge contains at least 10 weight percent of silicon, preferably more than 15 weight percent and most preferably more than 20 weight percent. However, the content may be lower and may, for example, go down to around 1 weight percent but is preferably above 2 weight percent. These low amounts of silicon are preferably used for slow charges or in situations where other fuel is incorporated, such as zirconium. The amount of dibismuth trioxide should exceed 30 weight percent, preferably exceeding 40 weight percent and more preferably exceeding 50 weight percent.

Over and above these obligatory components, other reactive and/or inert pyrotechnic additives may be incorporated in order to modify the burn rate or otherwise influence the reaction properties. Similarly, these additives should not give rise to gas releases. Examples of additives include fuels such as zirconium and boron or alternative oxidants such as iron oxide and manganese oxide or more inert components such as silicon oxide and titanium oxide.

The amount of such reactive additives is normally selected so that the total fuel/oxidant relationship falls within the above indicated range. The total amount of additives should not exceed 55 weight percent, preferably not exceeding 45 weight percent and more preferably under 30 weight percent.

Zirconium is a preferred alternative fuel, which provides, inter alia, enhanced ignitability and increased reaction rate. The amount may vary within wide limits, principally depending on the desired speed of the charge and may, for exmple, be between 1 and 50 weight percent, especially between 3 and 25 weight percent. Slow charges may have a content of between 1 and 20 weight percent, especially between 3 and 15 weight percent. Fast charges may, for example, have a content between 3 and 50 weight percent, especially between 5 and 25 weight percent. Primers and ignition charges may have a high content, for example exceeding 25 weight percent.

Additives other than pyrotechnic additives may also be incorporated in the charge, for example to improve the properties of the powder in relation to free flow and compactability, or binder additives to improve coherency or to allow granulation, for example clay minerals such as bentonite or carboxymethyl cellulose. The amounts of these sorts of additive are generally kept minor, for example below 4 weight percent, preferably below 2 weight percent and even more preferably under 1 weight percent. The lower of these limits appropriately apply to gas-releasing additives of this type, or are appropriate to gas-releasing additives in general, such as organic additives but also to inorganic additives such as chlorates.

The charges, in the usual manner, are preferably in the form of powder mixtures. The particle size may be used to influence the burn rate. The particle size of the incorporated main components, expressed as a weight average, may be between 0.1 and 100 microns, preferably between 1 and 50 microns. These values may also be appropriate for other optional pyrotechnic powder additives. The powder components or preferably the powder mixture may be granulated in order, for example, to facilitate dosing and compression.

The charges are relatively insensitive to unintended initiation and may be mixed and prepared in the dry state. It is preferred, however, that this is effected in the liquid state. The liquid may be an organic solvent but aqueous media and especially pure water are preferred because the components are water-insensitive. The mixture may be granulated from the liquid phase.

The charges may, as has been indicated, be used for all sorts of pyrotechnic applications, such as ignition charges, start charges etc. but preferably as delay charges, especially in civil detonators. In this connection, the charges are placed in the form of a layer directly in a detonator housing or are accomodated as a column in a surrounding housing element which is inserted into the detonator housing. The charge is placed between a component ignition device, for example a detonating cord, a low energy fuse (for instance Nonel, registered trade mark) or an electrically activated fuse head, and a functional main charge, usually a base charge of secondary explosive. The charge has sufficient initiation ability to be ignited by conventional ignition devices even without a special preceding primer, although these may be used if so desired. In the outward end, the charge may be allowed to act on a primary explosive, optionally via a transfer charge, or to directly ignite a secondary explosive, for example in the primary explosive-free detonator of the type apparent in Swedish patent application nos. 8404208-4 or 8803683-5, which are specifically incorporated herein by reference.

The above charges are generally press compacted.

The exact pressure of the press varies with the length of the charge, the form of the element etc. Appropriate end-densities may be within 10 and 80 percent of the crystal density of the mixture, especially between 20 and 60 percent of the crystal density.

The invention will be further exemplified with the following preferred but non-limiting embodiments.

Examples

A series of test charges was manufactured in accordance with the Examples below. The grain sizes of the incorporated components were determined prior to admixture with the "Fisher Sub Seive Sizer" method. Admixing of the charges was effected in aqueous phase (c. 40 - 50 weight percent water) with minor amounts of CMC as binder. The order of admixture was: dispersal of the bismuth oxide, addition of the binder in solution form, successive additions of the silicon powder and lastly addition of other, optional components to the mixture. Admixture was effected with the intensive mixer method. After admixture, the charges were oven-dried on trays to a moisture content of around 7 to 10 weight percent, after which granulation was effected on a seive cloth having a 0.8 mm mesh size, following which the granules were dried to a moisture content below 0.1 weight percent.

The charges were compressed with a pressure of about 1000 kp/cm$^2$ in delay elements of aluminium with an inner diameter of 3 mm and a length of 20 mm. The elements were inserted into detonators of the primary explosive containing type as well as the primary explosive-free type and were initiated with a low energy fuse of the Nonel (registered trade mark) type.

The figures indicated below for burn rates are based upon delay periods measured for at least 10 of such detonators for each charge. Elements have also been subjected to storage in humid and warm environments (+40°C and 75% relative humidity). These elements were then inserted into detonators and test-fired as above and showed to have maintained completely satisfactory functions and only insignificantly altered burn rates.

Example 1

A test charge was prepared in accordance with the following specification in which the percentages relate to weight percent and the particle sizes relate to average particle diameter:

28 % Si (silicon), particle size 3 μm
5 % Zr (zirconium), particle size 2 μm
67 % $Bi_2O_3$ (dibismuth trioxide), particle size 5 μm

The burn rate was measured as 76 mm/second.

Example 2

A test charge was prepared in accordance with the following specification in which the percentages relate to weight percent and the particle sizes relate to average particle diameter:

30 % Si (silicon), particle size 3 μm
20 % Zr (zirconium), particle size 2 μm
50 % $Bi_2O_3$ (dibismuth trioxide), particle size 5 μm

The burn rate was measured as 100 mm/second.

Example 3

A test charge was prepared in accordance with the following specification in which the percentages relate to weight percent and the particle sizes relate to average particle diameter:

40 % Si (silicon), particle size 3 μm
60 % $Bi_2O_3$ (bismuth trioxide), particle size 5 μm

The burn rate was measured as 35 mm/second.

Example 4

A test charge was prepared in accordance with the following specification in which the percentages relate to weight percent and the particle sizes relate to average particle diameter:

30 % Si (silicon), particle size 5 μm
20 % MnO (manganese oxide) particle size 4 μm
50 % $Bi_2O_3$ (dibismuth trioxide), particle size 5 μm

The burn rate was measured as 20 mm/second.

Example 5

A test charge was prepared in accordance with the following specification in which the percentages relate to weight percent and the particle sizes relate to average particle diameter:

32 % Si (silicon), particle size 3 μm
60 % $Bi_2O_3$ (dibismuth trioxide), particle size 5 μm
8 % $SiO_2$ (silicon dioxide), particle size < 1μm

The burn rate was measured as 11 mm/second.

Example 6

A test charge was prepared in accordance with the following specification in which the percentages relate to weight percent and the particle sizes relate to average particle diameter:

3 % Si (silicon), particle size 3 µm
10 % Zr (zirconium) particle size 2 µm
60 % $Bi_2O_3$ (dibismuth trioxide), particle size 5 µm
27 % $TiO_2$ (titanium dioxide), particle size < 1 µm

The burn rate was measured as 9 mm/second.

Example 7

A test charge was prepared in accordance with the following specification in which the percentages relate to weight percent and the particle sizes relate to average particle diameter:

5 % Si (silicon), particle size 3 µm
8 % Zr (zirconium) particle size 2 µm
62 % $Bi_2O_3$ (dibismuth trioxide), particle size 5 µm
25 % $TiO_2$ (titanium dioxide), particle size < 1 µm

The burn rate was measured as 7 mm/second.

Example 8

A test charge was prepared in accordance with the following specification in which the percentages relate to weight percent and the particle sizes relate to average particle diameter:

3 % Si (silicon), particle size 3 µm
97 % $Bi_2O_3$ (dibismuth trioxide), particle size 5 µm

The burn rate was measured as 5 mm/seconds.

**Claims**

1. A non-toxic and water-insoluble pyrotechnic delay charge for providing stable and reproducible delays in the millisecond and second ranges, **characterized in that** it comprises the components dibismuth trioxide as an oxidation agent and silicon (Si) as a fuel, the amount of dibismuth trioxide being more than 30 % by weight.

2. A charge according to claim 1, **characterized in that** it comprises more than 2 weight percent of silicon.

3. A charge according to claim 2, **characterized in that** it comprises more than 15 weight percent of silicon.

4. A charge according to any one of the preceding claims, **characterized in that** it comprises an additive of other reactive and/or inert pyrotechnic components in an amount of no more than 55 % by weight.

5. A charge according to claim 4, **characterized in that** the additive comprises zirconium (Zr).

6. A charge according to claim 5, **characterized in that** the amount of zirconium is between 1 and 47 % by weight of the charge.

7. A charge according to claim 6, **characterized in that** the amount of zirconium is between 3 and 25 % by weight of the charge.

8. A charge according to any one of the preceding claims, **characterized in that** it has a stoichiometric excess of fuel.

9. A charge according to any one of the preceding claims, **characterized in that** it contains a binder in an amount of no more than 4 % by weight.

10. A charge according to claim 9, **characterized in that** the binder comprises carboxymethyl cellulose.

11. A charge according to any one of the preceding claims, **characterized in that** the components are in the form of a powder with particle sizes between 0.1 and 100 µm, expressed as weight averages.

12. A charge according to claim 11, **characterized in that** the components or the charge have been granulated.

13. A charge according to any one of the preceding claims, **characterized in that** it has a burn rate between 1 and 20 mm/s.

14. A charge according to any one of claims 1 to 12, **characterized in** that it has a burn rate between 10 and 200 mm/s.

15. A charge according to any one of the preceding claims, **characterized in that** it has a density between 20 and 60 percent of the crystal density of the mixture.

16. A pyrotechnic delay element for providing a delay in the millisecond and second ranges, **characterized in** that it comprises an enclosure accomodating a delay charge as defined in any one of claims 1-15.

17. An element according to claim 16, **characterized in that** the enclosure is constituted by a detonator housing.

18. An element according to claim 16 or 17, **characterized in that** the enclosure comprises a substantially cylindrical metal casing.

19. An element according to any one of claims 16-18, **characterized in that** the charge is substantially

cylindrical.

20. An element according to claim 19, **characterized in that** the charge diameter is between 1 and 10 mm.

21. An element according to claim 19 or 20, **characterized in that** the charge length is between 1 and 100 mm, especially between 2 and 50 mm.

22. A detonator comprising a housing, ignition means disposed at one end of the housing, a base charge of a secondary explosive disposed at the other end of the housing and a pyrotechnic delay charge disposed therebetween, **characterized in that** the delay charge is as defined in any one of claims 1-15.

**Patentansprüche**

1. Nicht-toxische und wasserunlösliche pyrotechnische Verzögerungsladung zum Bereitstellen stabiler und reproduzierbarer Verzögerungen in den Millisekunden- und Sekundenbereichen,
**dadurch gekennzeichnet**, daß
sie umfaßt die Bestandteile Dibismuttrioxid als ein Oxidationsmittel und Silicium (Si) als Brennstoff, wobei der Gehalt an Dibismuttrioxid mehr als 30 Gew.-% ausmacht.

2. Ladung nach Anspruch 1,
**dadurch gekennzeichnet**, daß
sie mehr als 2 Gew.-% an Silicium umfaßt.

3. Ladung nach Anspruch 2,
**dadurch gekennzeichnet**, daß sie mehr als 15 Gew.-% an Silicium umfaßt.

4. Ladung nach einem der vorangegangenen Ansprüche,
**dadurch gekennzeichnet**, daß
sie ein Additiv umfaßt an anderen reaktiven und/oder inerten pyrotechnischen Komponenten bzw. Bestandteilen, bei einem Gehalt von nicht mehr als 55 Gew.-%.

5. Ladung nach Anspruch 4,
**dadurch gekennzeichnet**, daß
das Additiv Zirkon (Zr) umfaßt.

6. Ladung nach Anspruch 5,
**dadurch gekennzeichnet**, daß
der Gehalt an Zirkon zwischen 1 und 47 Gew.-% der Ladung beträgt.

7. Ladung nach Anspruch 6,
**dadurch gekennzeichnet**, daß
der Gehalt an Zirkon zwischen 3 und 25 Gew.-% der Ladung liegt.

8. Ladung nach einem der vorangegangenen Ansprüche,
**dadurch gekennzeichnet**, daß
sie einen stoichiometrischen Überschuß an Brennstoff enthält.

9. Ladung nach einem der vorangegangenen Ansprüche,
**dadurch gekennzeichnet**, daß
sie ein Bindemittel enthält bei einem Gehalt bzw. Ausmaß von nicht mehr als 4 Gew.-%.

10. Ladung nach Anspruch 9,
**dadurch gekennzeichnet**, daß
das Bindemittel Carboxymethylcellulose umfaßt.

11. Ladung nach einem der vorangegangenen Ansprüche,
**dadurch gekennzeichnet**, daß
die Komponenten bzw. Bestandteile in der Form eines Pulvers vorliegen, mit Partikelgrößen zwischen 0,1 und 100 µm, ausgedrückt als Gewichtsmittelwerte.

12. Ladung nach Anspruch 11,
**dadurch gekennzeichnet**, daß
die Bestandteile bzw. Komponenten oder die Ladung granuliert wurden.

13. Ladung nach einem der vorangegangenen Ansprüche,
**dadurch gekennzeichnet**, daß
sie eine Brennrate bzw. -geschwindigkeit zwischen 1 und 20 mm/s aufweist.

14. Ladung nach einem der Ansprüche 1 bis 12,
**dadurch gekennzeichnet**, daß
sie eine Brennrate zwischen 10 und 200 mm/s aufweist.

15. Ladung nach einem der vorangegangenen Ansprüche,
**dadurch gekennzeichnet**, daß
sie eine Dichte zwischen 20 und 60 Prozent der Kristalldichte des Gemisches aufweist.

16. Pyrotechnisches Verzögerungselement zum Bereitstellen einer Verzögerung in den Millisekunden- und Sekundenbereichen,
**dadurch gekennzeichnet**, daß
es umfaßt eine Umhüllung, aufnehmend eine Verzögerungsladung nach einem der Ansprüche 1 bis 15.

17. Element nach Anspruch 16,
**dadurch gekennzeichnet**, daß

die Umhüllung durch ein Detonatorgehäuse gebildet ist.

18. Element nach Anspruch 16 oder 17, **dadurch gekennzeichnet**, daß die Umhüllung ein im wesentlichen zylindrisches Metallgehäuse umfaßt.

19. Element nach einem der Ansprüche 16 bis 18, **dadurch gekennzeichnet**, daß die Ladung im wesentlichen zylindrisch ist.

20. Element nach Anspruch 19, **dadurch gekennzeichnet**, daß der Ladungsdurchmesser zwischen 1 und 10 mm beträgt.

21. Element nach Anspruch 19 oder 20, **dadurch gekennzeichnet**, daß die Ladungslänge zwischen 1 und 100 mm, insbesondere zwischen 2 und 50 mm, beträgt.

22. Detonator, umfassend ein Gehäuse, eine Zündeinrichtung, angeordnet an einem Ende des Gehäuses, eine Grundladung eines Sekundärsprengstoffes, angeordnet an dem anderen Ende des Gehäuses, und eine pyrotechnische Verzögerungsladung, welche dazwischen angeordnet ist, **dadurch gekennzeichnet**, daß die Verzögerungsladung eine nach einem der Ansprüche 1 bis 15 ist.

## Revendications

1. Charge à retard pyrotechnique non toxique et insoluble dans l'eau pour fournir des délais stables et reproductibles de l'intervalle de la milliseconde et de la seconde, caractérisée en ce qu'elle comprend les composants trioxyde de dibismuth comme agent d'oxydation et silicium (Si) comme combustible, la quantité de trioxyde de dibismuth étant supérieure à 30% en poids.

2. Charge selon la revendication 1, caractérisée en ce qu'elle comprend plus de 2% en poids de silicium.

3. Charge selon la revendication 2, caractérisée en ce qu'elle comprend plus de 15% en poids de silicium.

4. Charge selon l'une quelconque des revendications précédentes, caractérisée en ce qu'elle comprend un additif d'autres composants pyrotechniques réactifs et/ou inertes en une quantité ne dépassant pas 55% en poids.

5. Charge selon la revendication 4, caractérisée en ce que l'additif comprend du zirconium (Zr).

6. Charge selon la revendication 5, caractérisée en ce que la quantité de zirconium est comprise entre 1 et 47% en poids de la charge.

7. Charge selon la revendication 6, caractérisée en ce que la quantité de zirconium est comprise entre 3 et 25% en poids de la charge.

8. Charge selon l'une quelconque des revendications précédentes, caractérisée en ce qu'elle comporte un excès stoechiométrique de combustible.

9. Charge selon l'une quelconque des revendications précédentes, caractérisée en ce qu'elle contient un liant en une quantité ne dépassant pas 4% en poids.

10. Charge selon la revendication 9, caractérisée en ce que le liant comprend de la carboxyméthyl-cellulose.

11. Charge selon l'une quelconque des revendications précédentes, caractérisée en ce que les composants sont sous la forme d'une poudre ayant des tailles particulaires comprises entre 0,1 et 100 µm, exprimées en moyennes pondérales.

12. Charge selon la revendication 11, caractérisée en ce que les composants ou la charge ont été granulés.

13. Charge selon l'une quelconque des revendications précédentes, caractérisée en ce qu'elle présente une vitesse de combustion comprise entre 1 et 20 mm/s.

14. Charge selon l'une quelconque des revendications 1 à 12, caractérisée en ce qu'elle présente une vitesse de combustion comprise entre 10 et 200 mm/s.

15. Charge selon l'une quelconque des revendications précédentes, caractérisée en ce qu'elle a une densité comprise entre 20 et 60% de la densité cristalline du mélange.

16. Elément de retard pyrotechnique pour fournir un retard de l'intervalle de la milliseconde et de la seconde, caractérisé en ce qu'il comprend une enceinte abritant une charge à retard telle que définie dans l'une quelconque des revendications 1-15.

17. Elément selon la revendication 16, caractérisé en ce que l'enceinte est constituée par une enveloppe de détonateur.

18. Elément selon la revendication 16 ou 17, caractérisé en ce que l'enceinte comprend un boîtier en

métal sensiblement cylindrique.

19. Elément selon l'une quelconque des revendications 16-18, caractérisé en ce que la charge est sensiblement cylindrique.

20. Elément selon la revendication 19, caractérisé en ce que le diamètre de la charge est compris entre 1 et 10 mm.

21. Elément selon la revendication 19 ou 20, caractérisé en ce que la longueur de la charge est comprise entre 1 et 100 mm, en particulier entre 2 et 50 mm.

22. Détonateur comprenant une enveloppe, un organe d'allumage dispose à une extrémité de l'enveloppe, une charge de base d'un explosif secondaire disposée à l'autre extrémité de l'enveloppe et une charge à retard pyrotechnique disposée entre eux, caractérisé en ce que la charge à retard est telle que définie dans l'une quelconque des revendications 1-15.